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Final Scientific Report

AFOSR 78-3563

X-RAY OPTICS

Period of report: February 1, 1979 to March 31, 1981

Prepared for

Electronics and Material Sciences

Air Force Office of Scientific Research

Bolling Air Force Base, D.C. 20332

Report submitted by

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X-RAY OPTICS

1.0 SCOPE

This final scientific report ~~on AFOSR~~ 78-3563 covers research activities on X-Ray Optics during the period from 1 February 1979 to 31 March 1981. It is an updated revision of an earlier interim report which covered the period from 1 February 1979 to 31 January 1980. The report description is contained in the following sections:

- 2.0 Preliminary Study of Source Coherency
- 3.0 Lloyd's Mirror Experiment
- 4.0 X-Ray Speckle
  - 4.1 Measurement of Film Resolution
  - 4.2 Experiments of 20 - 22 May 1980
  - 4.3 Experiments of 8 September 1980
  - 4.4 Conclusions and Plans
- 5.0 Personnel

2.0 PRELIMINARY STUDY OF SOURCE COHERENCY

Data were obtained from Professor K. Das Gupta's apparatus using an intense micro-focused electron beam to generate x-rays. A representative sample is shown in Fig. 2.1. These patterns were obtained with a single slit of less than 0.5 μm using the layout shown in Fig. 2.2. The most evident aspect of this pattern is the step-like character running collinearly with the slit.

Two main questions regarding these data can be posed.

1) What causes the step-like structure?
2) Is there any evidence of speckle in this pattern?

-2 -1 0 +1 +2

(a) Single Slit (less than 0.5μm) interference pattern with 1.54Å copper x-rays (non-linear) operation at 13 KV, 0.5 mA. Slit to film 60 cm, source to slit 5 cm.

-1 0 +1

(b) Same as in 2.1(a) above. Operation in linear regime with less prominent interference pattern.

Fig. 2.1

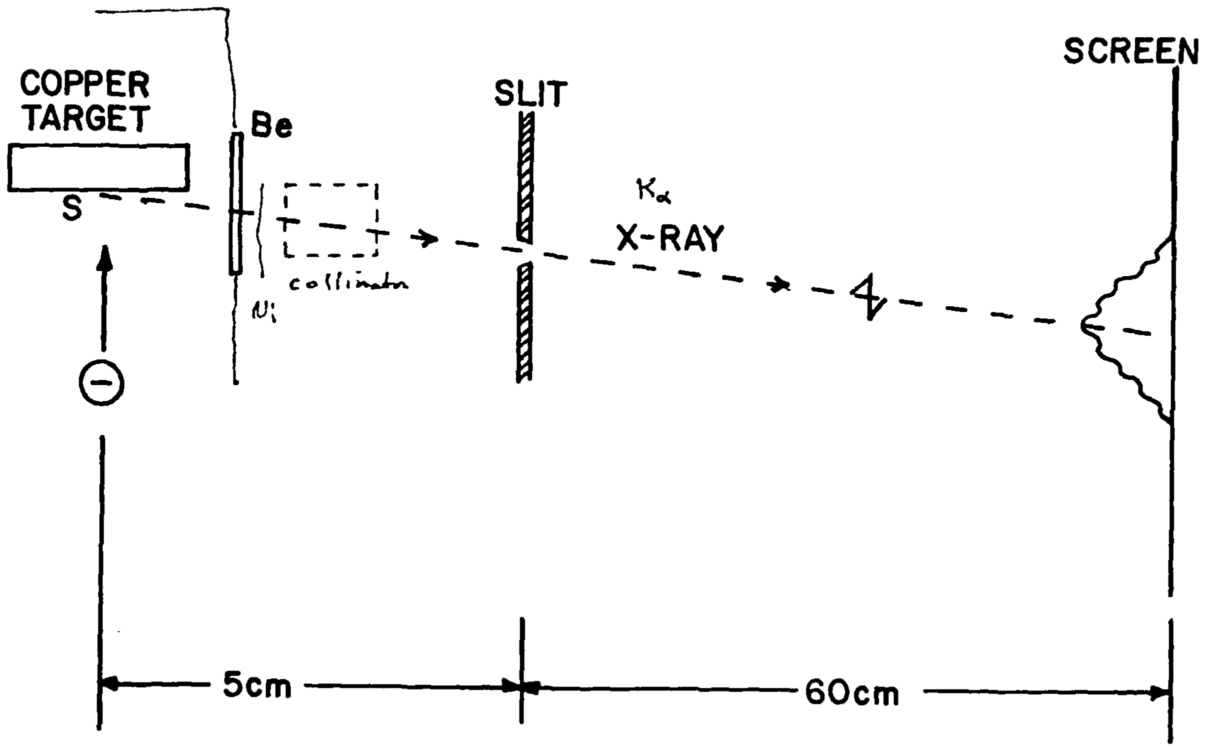

Fig. 2.2 Experimental configuration used by K. Das Gupta to obtain records of Fig. 2.1. Microfocussed electron beam S approximately 30 μm by 100 μm. Slit width at 0.5 μm (approx.).

Several careful densitometer traces were made at The Institute of Optics. A typical curve is shown in Fig. 2.3.

One readily observes three main features of this pattern. One is the general bell-like shape of the entire envelope. Second is the coarse stepwise pattern that is also evident in the photographs. Thirdly, we note the fine scale ripple on the pattern.

Several preliminary conclusions can be drawn from this curve. If the radiation were nearly monochromatic, there should be very sharp interference lobes. The dependence should be like the sinc (x) function. If the radiation is partially coherent, then it is to be expected that the lobes would fill-in. The pattern has the appearance typical of that from a partially coherent, extended source or aperture (30 μm x 100 μm) which illuminates a narrow slit. This pattern can be calculated using the formalism and approach of Shore, Thompson and Whitney.[+] One can readily see the similarities between Fig. 5 of the reference and our Fig. 2.3. However, we find that in order to make a definitive comparison and determination of the coherency parameters, one would have

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[+] "Diffraction by Apertures Illuminated with Partially Coherent Light," R.A. Shore, B.J. Thompson, and R.E. Whitney, J. Opt. Soc. Am. 56, pp 733-738 (1966).

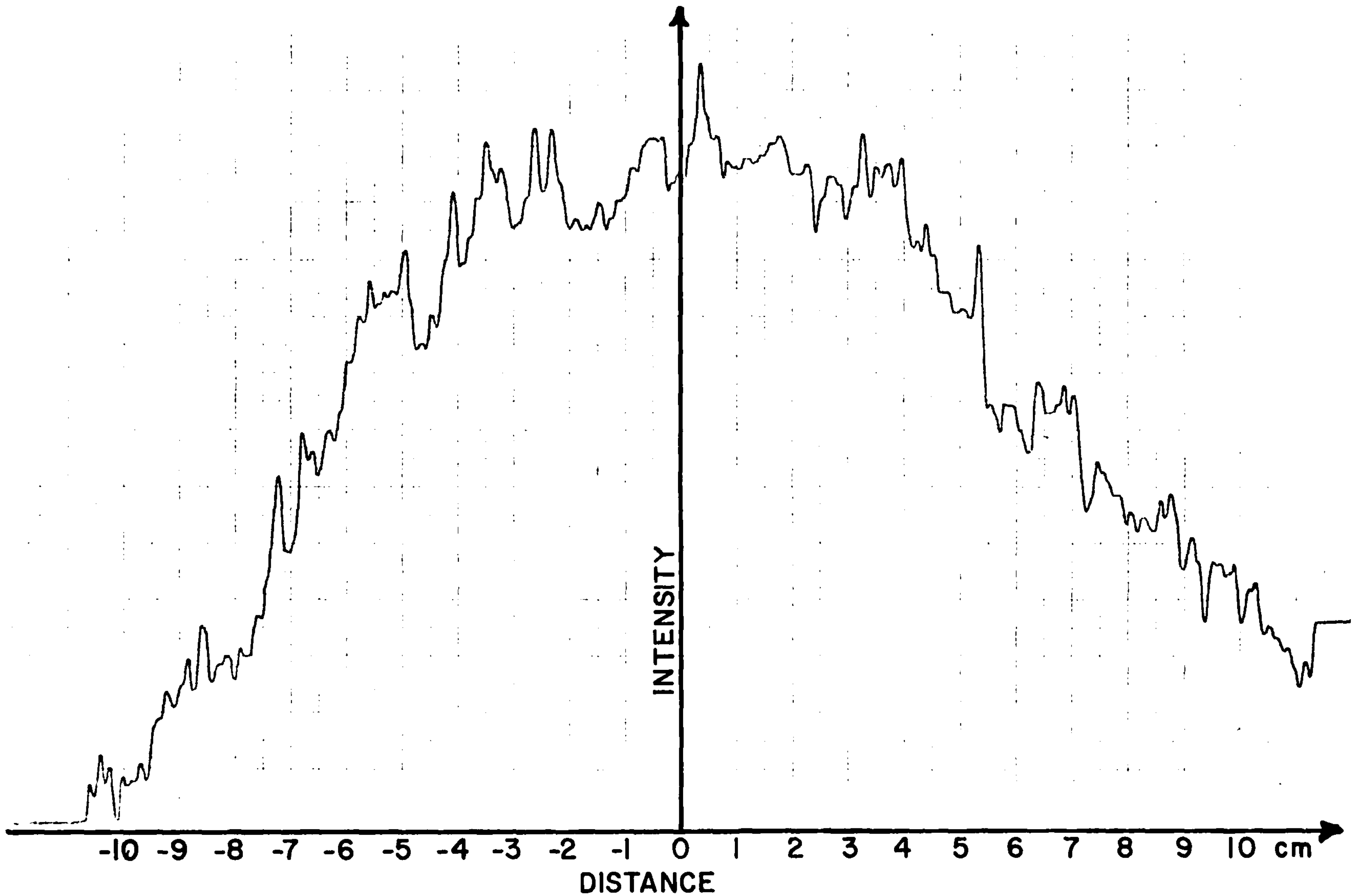

Fig. 2.3. Intensity vs. distance for x-ray recorded in setup of Fig. 2.2. Data are taken from photograph in Fig. 2.1(a) with 9 times magnification of distance.

to make a series of measurements as the slit width is varied. Professor K. Das Gupta has agreed to work with us on making this set of measurements during the remaining phase of this investigation.

From our viewpoint, the most interesting feature of Fig. 2.3 is the fine scale ripple. Our preliminary conclusion is that this is a manifestation of x-ray speckle. In order to make a definitive conclusion in this regard, there are two main features of any speckle pattern which need to be studied. One is its spatial size and the other is contrast. For the dimensions used in the experiment, the speckle size d should be given by

$$d = \frac{\lambda}{w} L s,$$

where λ is the wavelength, w is the slit width, L is the distance from the slit to the screen, and s is the scaling or magnification used in the microdensitometer. For $\lambda = 1.54\mathring{A}$, $w = 0.5\ \mu m$, $L = 60$ cm, and $s = 9.0$, we find the speckle size d to be

$$d = 1.66 \text{ mm}.$$

From Fig. 2.3, it is seen that an average size of 2. mm is an excellent estimate.

The next step in this research is to study data as the slit size is varied. As is seen in the equation above, larger slit width w leads to a ripple of finer scale. This is an extremely important experiment, and the improved monochromaticity (if that is what is happening) is only obtained with Prof. K. Das Gupta's apparatus. Unfortunately, this apparatus had not been functioning for about two months due to a technical problem in the vacuum system. This has been corrected and we are hopeful that further data will be available before March 15.

The contrast of the speckle (temporary label) is relatively low. This can be caused by two main factors:

a) Very little roughness in the propagation path, including the surface structure of the x-ray emitter

b) Temporal bandwidth of the source is large

One can estimate the linewidth of the source and the effective roughness by a study of the speckle contrast. Once the source becomes too monochromatic, the contrast becomes large and a roughness must be introduced artificially in order to measure linewidths in this manner. From the outset, this was one of our major goals, and so it will be emphasized during the next several months.

For a fixed transmission system, the increased contrast or increased spike-like character of the intensity is a positive indication of a narrower line-width. Measurements of this type can be classed under (b) above.

If one wants to measure very slight roughness, on the order of tens of Angstroms, then the speckle contrast is measured with a fixed source and a variable transmission path, e.g., the x-ray is transmitted through a substrate or reflected at grazing incidence. Experiments are classed as type (a).

Again from the data at hand, we have a preliminary conclusion. A densitometer trace of Fig. 1(b) is shown in Fig. 2.4. We note the greatly lessened speckle fluctuation in this trace (in comparison to that shown in Fig. 2.3). From this we conclude that the linewidth is somewhat narrower in Fig. 1(a) than it is in Fig. 1(b).

During the next period of this research, we plan to make quantitative assessments of this method of speckle applied to the x-ray regime. It should be useful both for making measurements of fine-scale roughness or for making linewidth measurements of very narrow band sources.

3.0 LLOYD'S MIRROR EXPERIMENT

In Section 3.1 we describe the experimental plan for this phase of the research and in Section 3.2 we describe the progress.

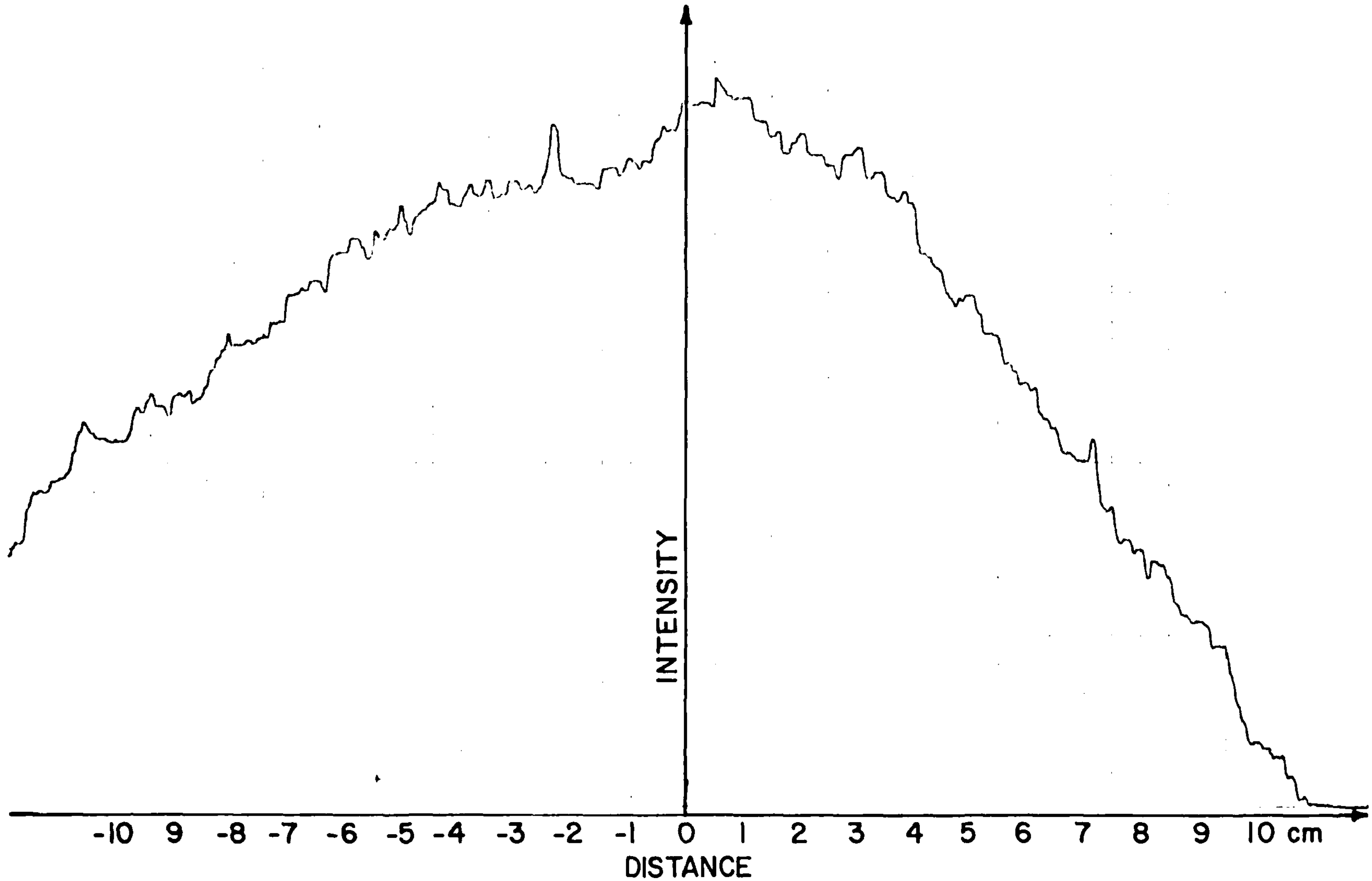

Fig. 2.4. Intensity vs. distance for x-ray recorded in setup of Fig. 2.2. Data are taken from photograph in Fig. 2.1(b) with 9 times magnification of distance.

3.1 Experimental Plan

In this experiment, shown in Fig. 3.1 the effective position of the point source of x-ray at S is incident at small angles, grazing the highly polished aluminum mirror. The reflection and the direct beam overlap on the viewing screen for the portion x_1 to x_2 along the x-axis which is collinear with the normal from S to the plane of M. This experiment has numerous advantages at x-ray wavelengths, as well as non-trivial difficulties so the reader may choose to consider it as hypothetical at this stage. A detailed consideration now does serve to illustrate the research objectives in a simple clear fashion, giving accurate numerical constraints, without unnecessary experimental detail.

At a position x on the recording medium, the path difference Δs between the direct ray from S and the reflected ray from S' is given approximately by

$$\Delta s = \frac{2xd}{L} \quad . \tag{1}$$

The source to image spacing is d and the total distance is $L = L_1 + L_2$ where L_2 is the nearest distance from the mirror to the screen. The ratios d/L and x/L are assumed much less than unity.

The temporal coherence τ_c of the point source S is characterized by its spectral linewidth $\Delta\nu_c$, related by

$$\tau_c = \frac{1}{\Delta\nu_c} \quad . \tag{2}$$

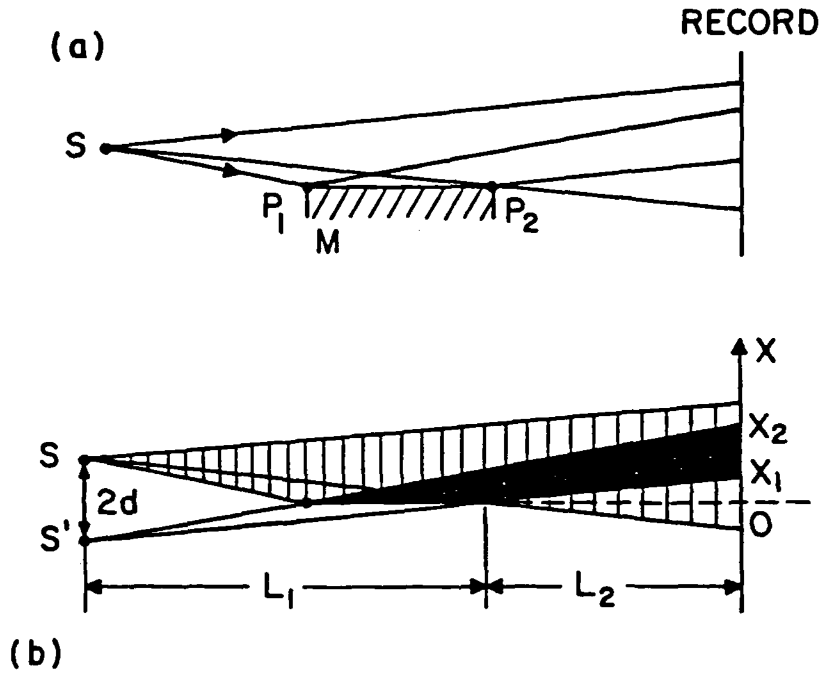

Fig. 3.1 Lloyd's mirror experiment: (a) with a point source at S, a mirror M and film or other recording medium and (b) equivalent point image showing the overlap or interference region extends from $0<X_1$ to X_2.

The coherence length L_c is generally defined as

$$L_c = c\tau_c$$
$$L_c = \frac{c}{\Delta\nu_c} = \frac{\lambda^2}{\Delta\lambda_c} \quad , \tag{3}$$

when $\Delta\lambda_c/\lambda << 1$.

The path distance Δs in Eq. (1) gives the fringe spacing along x, by setting the difference $2\ \Delta x\ d/L$ equal to a wavelength. Hence, the fringe spacing is

$$\Delta x = \frac{\lambda L}{2d} \tag{4}$$

However, the contrast in the fringe pattern depends upon the degree of coherence in the source. To understand the subtleties of this contrast variation, one needs to apply the analytical methods in the theory of partical coherence.[1] It is well known that the contrast C_R in the fringe pattern is high when $\Delta s \approx 0$, and it will fall off sharply as $\Delta s \to L_c$. This is the basis for measuring $\Delta\nu_c$[2].

We measure x at which C_R has dropped to a threshold value, and by Eqs. (1) and (3) we compute

$$\frac{\Delta\lambda_c}{\lambda^2} = \frac{L}{2x_c d} \tag{5}$$

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[1] M. Born and E. Wolf: "Principles of Optics", Pergamon Press, Fourth Edition (1970), p. 491.

[2] B.J. Thompson and E. Wolf, J. Opt. Soc. Amer., 47 (1957), 895.

A. Wavelengths of interest

X-ray holography at several different wavelengths is of basic interest, since the pictures can be expected to vary appreciably with an order of magnitude change in wavelength. Many of our experiments, in cooperation with K. Das Gupta,[3] use his laser-like, narrowed, intense x-ray radiation at 1.5406 Å.

Other experiments have been conducted at 44.8 Å in the work of Reuter and Mahr[4] (Cornell - 1976) and Aoki and Kikuta[5] (Tokyo University - 1974). S.J. Burns of the University of Rochester and his coworkers at the University of Colorado have studied the production of high intensity, monochromatic, spatially coherent x-rays (1974) and their method has been studied.[6]

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3 See Table I, in particular, items 7 and 8.

4 B. Reuter and H. Mahr, "Experiments with Fourier transform holograms using 4.48 nm x-rays", J. Of Phys. E: Scientific Instruments 1976, vol. 9, 746.

5 S. Aoki and S. Kikuta, "X-ray Holographic Microscopy", Japanese J. Of Appl. Phys. 13, (1974) 1385.

6 S.J. Burns, W.L. Pickles, C.S. Barrett, J.B. Newkirk, and C.O. Ruud, Article in Advances in X-ray Analysis 18, 425-36 (1976).

Table I - Some useful x-ray wavelengths for interference experiments

Source	Å	Target
1. Copper, doubly monochromatized by dislocation from silicon crystal, using GE-sealed off tube	1.5406	Copper
	2.290	Chromium
	1.789	Cobalt
	2.749	Titanium
2. Mossbauer source, 10 or 12 wavelengths, no background, monochromatic but weak		
3. Iron-55 has 5-year half-life, low background	2.1018	
4. Aluminum K_α	8.32	
5. Carbon K_α [9]	44.8	
6. Be K_α [9]	114.	
7. Germanium, single crystal Kossel-Borrmann radiation pumped simultaneously by electrons and photons.[10]	1.5406	
8. Sapphire, Silicon, Mica. Again simultaneous bombardment to produce highly monochromatic x-ray.[11]	8 to 10	

[9] J.W. Giles, J. Opt. Soc. Am. 59 (1969) 1179. Recorded in-line hologram of a glass fiber 5.9 μm diameter.

[10] K. Das Gupta, "Diffraction of Discrete x-ray Frequencies from Germanium Atoms in Crystals", J. Appl. Phy. 47, 2765 (1976).

[11] K. Das Gupta, unpublished.

It should be emphasized that x-ray lines narrowed by cascaded Bragg reflection have adequate coherence for interference experiments. Using his two curved crystal spectrometer, Das Gupta reports an angular divergence of 10 arc sec and a linewidth on the order of 0.01 ev.[7] This is consistent with the divergence of 10^{-5} radians and linewidth $\Delta\lambda/\lambda$ of 0.8 x 10^{-5} reported for Copper $K_{\alpha 1}$ by Matsushita who also used a double silicon crystal monochromater.[8]

At wavelengths below 3 Å, atmospheric absorption is not troublesome; however, dimensional stability and media resolution are. Table II shows fringe spacings at 2 Å and 50 Å. A helium atmosphere may be useful between 2.5 Å and 10 Å, or vacuum; and at 44.8 Å vacuum operation is essential.

The resolution requirements for recording x-ray interference patterns in the holographic context are extreme. For the Lloyd's mirror experiment, the grazing angle is about 15 to 20 arc minutes. Any smaller value is fine, the reflectivity is very high, but it drops sharply above the critical value.

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[7] K. Das Gupta, X-ray Diffraction Camera Employing Two Curved Crystal Transmission Type Monochromaters, U.S. Pattent No. 3,379,876 (1968) and Phys. Letters 46A 179 (1973).

[8] T. Matsushita, J. Appl. Crystallogr. (Denmark) 7, 254-9, (1974).

3.2 Experiments

The experimental configuration is shown in Fig. 3.2. A tube with a copper target is used with an output collimator designed to mount close to the Beryillium window but at a slight angle so as to pass the most intense portion of the K_{α} line. A crystal filter spectrometer was assembled. Several shielding slits were used in order to pass the collimated beam but eliminate the $K_{\alpha 2}$ and $K_{\alpha 3}$ lines and also to reduce the stray radiation to a safe level. A series of metallic mirrors were used (M in Fig. 3.2) carefully adjusted for grazing angles of incidence. While several experiments were made, we have not succeeded in recording fringes with this method.

Refinements were made to the mirror alignment system. A laser beam was used, carefully collimated to the $K_{\alpha 1}$ line, so that visual adjustment of M could be made. Then a very precise rotary table with an angular resolution estimated at less than 10 arc seconds was installed as an additional refinement. While relatively good signal level was obtained in the film recording, still no fringe pattern was observed.

Unfortunately we were also plagued with several malfunctions of the x-ray source, including tube degredation, failure of the water cooling, and so on. At this point it was decided to rebuild the x-ray apparatus relatively extensively and to try a different type of interference experiment.

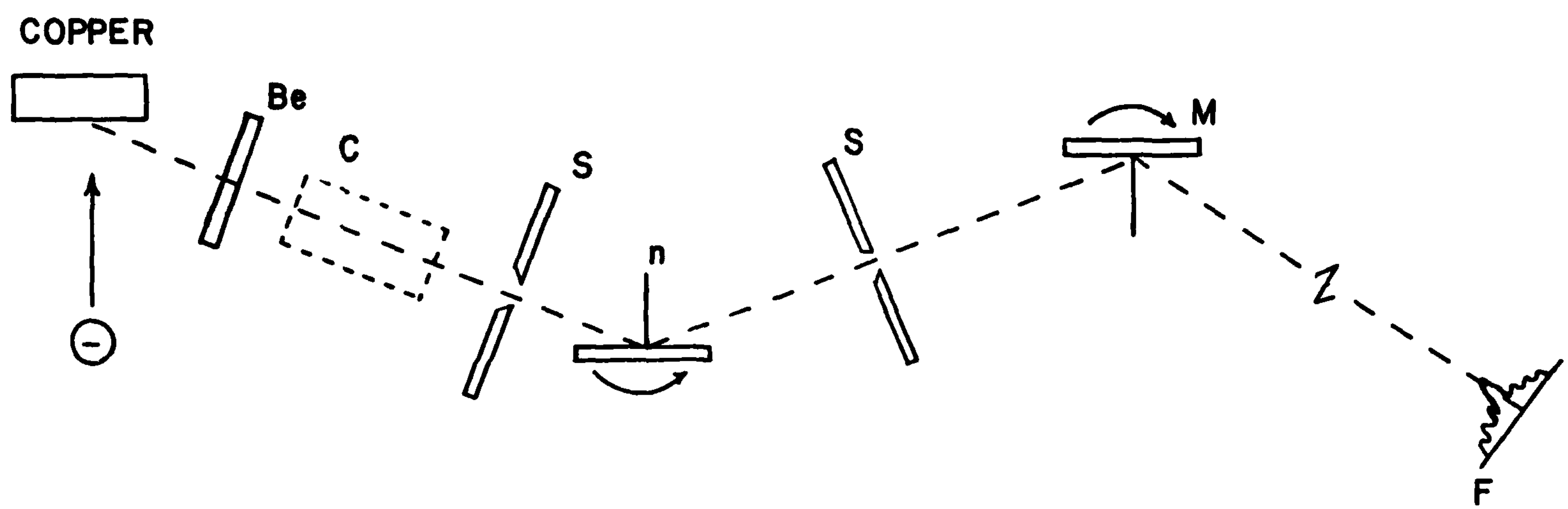

Fig. 3.2. Experimental setup for $K_{\alpha 1}$ line with collimator C, slits S, Lloyd's mirror M, and film recording F.

In this second approach we are fabricating a special multi-layer with a controlled wedge. This should provide two beams at a well-controlled angle and this will eliminate many of the difficulties we have experienced to date. The details of this new approach are described more fully in the following section.

4.0 X-RAY SPECKLE

In order to observe speckle phenomena, the radiation source must have sufficient coherence. A method to determine source coherence is to measure the fringe contrast in a two-beam interference experiment. During the preceeding contract period, experiments have been conducted using a Lloyd's mirror arrangement. The results and conclusions from this research are described in detail in Section 3.0. Briefly, in the experiments we found the positional requirements in angle to be quite stringent. To achieve the small path differences that are required to observe interference, angular positioning of approximately ± 10 arc seconds is required. With the existing apparatus, we were not able to achieve these tolerances, thus the interference, necessary to study beam coherence, was not observed. In the upcoming period, a new approach to obtain small path differences is being investigated. The method involves the introduction of a wedge to produce the interference pattern, instead of tiny grazing angles as required in the Lloyd's mirror experiment. The basic idea is illustrated in Fig. 4.1. A colimated x-ray beam is incident on the wedge structure. The structure consists of a quartz flat, a thick (approximately 1000 Å) layer of gold and an aluminum wedge with angle $\Delta\theta$.

On reflection, two beams are produced. One beam is propagating with direction angle θ(from the aluminum-gold interface); the other has a direction angle $\theta-2\Delta\theta$ (from the air-aluminum interface). A two-beam interference pattern should be produced at the film plane. For this arrangement, the fringe spacing is found to be

$$\Delta X = \frac{\lambda}{\sin(2\Delta\theta)} \qquad (4.1)$$

$$\Delta X \simeq \frac{\lambda}{2\Delta\theta} \quad .$$

Of course, the contrast of fringes depends on the coherence structure of the beam.[+] In this experiment, observation of fringes will demonstrate that the beam has the necessary coherence to produce a speckle pattern.

In the design of the aluminum wedge structure, one must consider the requirements for film resolution and

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[+] M. Born and E. Wolf, Principles of Optics, 5th ed (Pergamon, Oxford, 1975), Ch. 10.

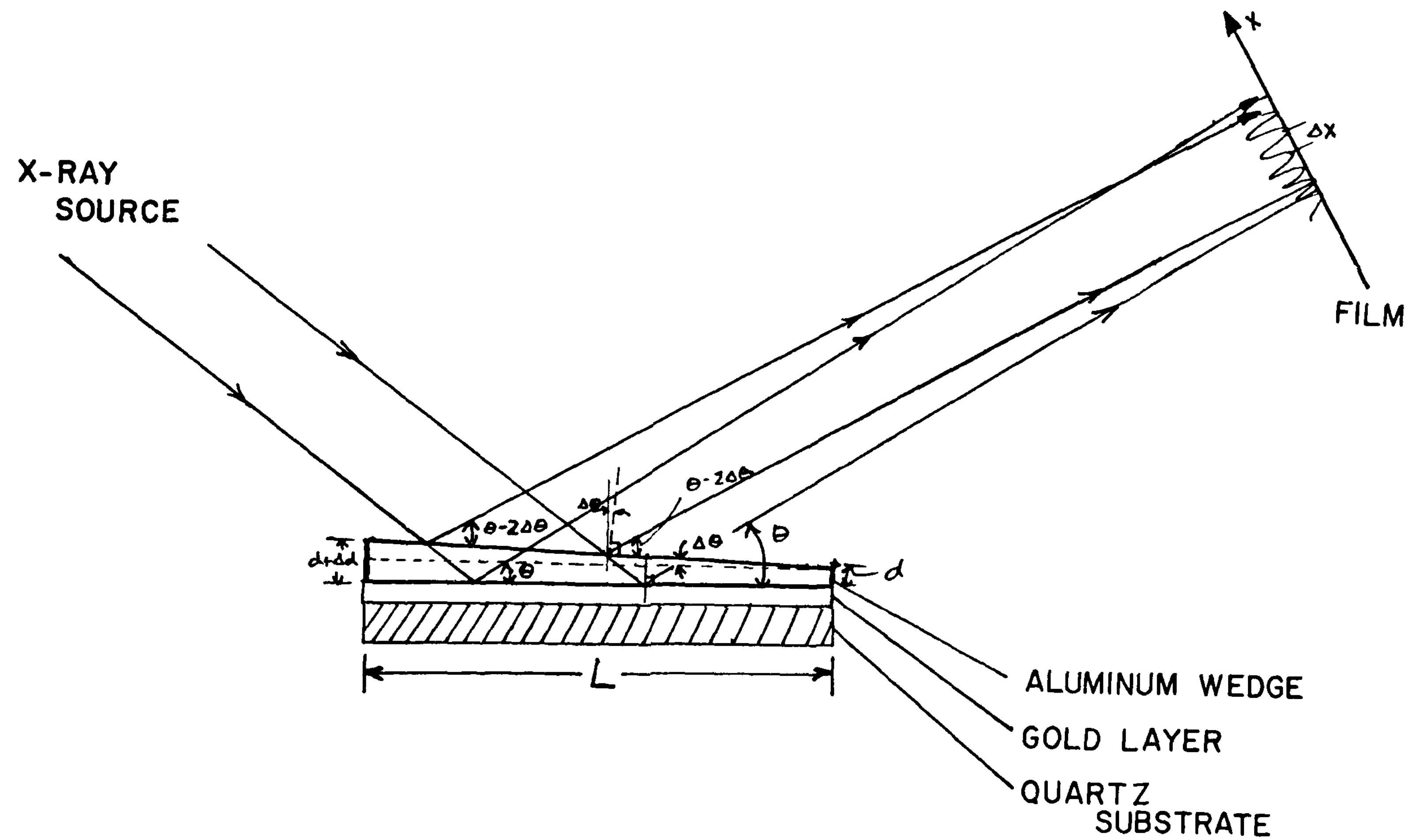

Fig. 4.1. Multilayer structure for two-beam interference equipment. A collimated x-ray beam is incident on the multilayer. On reflection, two beams are produced. One has direction angle $\theta-2\Delta\theta$ (air/aluminum interface), the other has direction angle θ (aluminum/gold interface). The reflected beams interfere, producing a fringe pattern at the film plane.

collimated beam diameter. These factors determine the value of the wedge angle $\Delta\theta$ that is needed. So as to not exceed the resolution requirements of available films, we will design the wedge to produce approximately 100 fringes in a millimeter. In our experiments, the diameter of the collimated beam is about one millimeter. Thus from Eq. (4.1) we need to fabricate a wedge with angle $\Delta\theta \simeq 7.7\times10^{-6}$ radians for the copper K_α line ($\lambda = 1.54\text{Å}$). This can be made by tipping the gold/quartz substrate at angle $\Delta\phi$ in the evaporation chamber, see Fig. 4.2.

As in the Lloyd's mirror experiment, the path difference between the two reflected beams must be kept small. A critical design parameter that affects path difference is the thickness, d, of the aluminum layer. We plan to make two wedges that have different values for d--one small, d = 50 Å; and the other larger, d = 150 - 200 Å.

A central goal in our research is to observe x-ray speckle. Upon verification of the source coherence from the two-beam interference experiments discussed above, the following speckle experiment, depicted in Fig. 4.3, will be performed.

First, we will roughen the surface of the aluminum wedge that was used to produce fringes. The roughness requirement to produce a well-developed speckle pattern is that the standard deviation of the rough surface be much greater than the wavelength of radiation, which in our case is 1.54 Å.

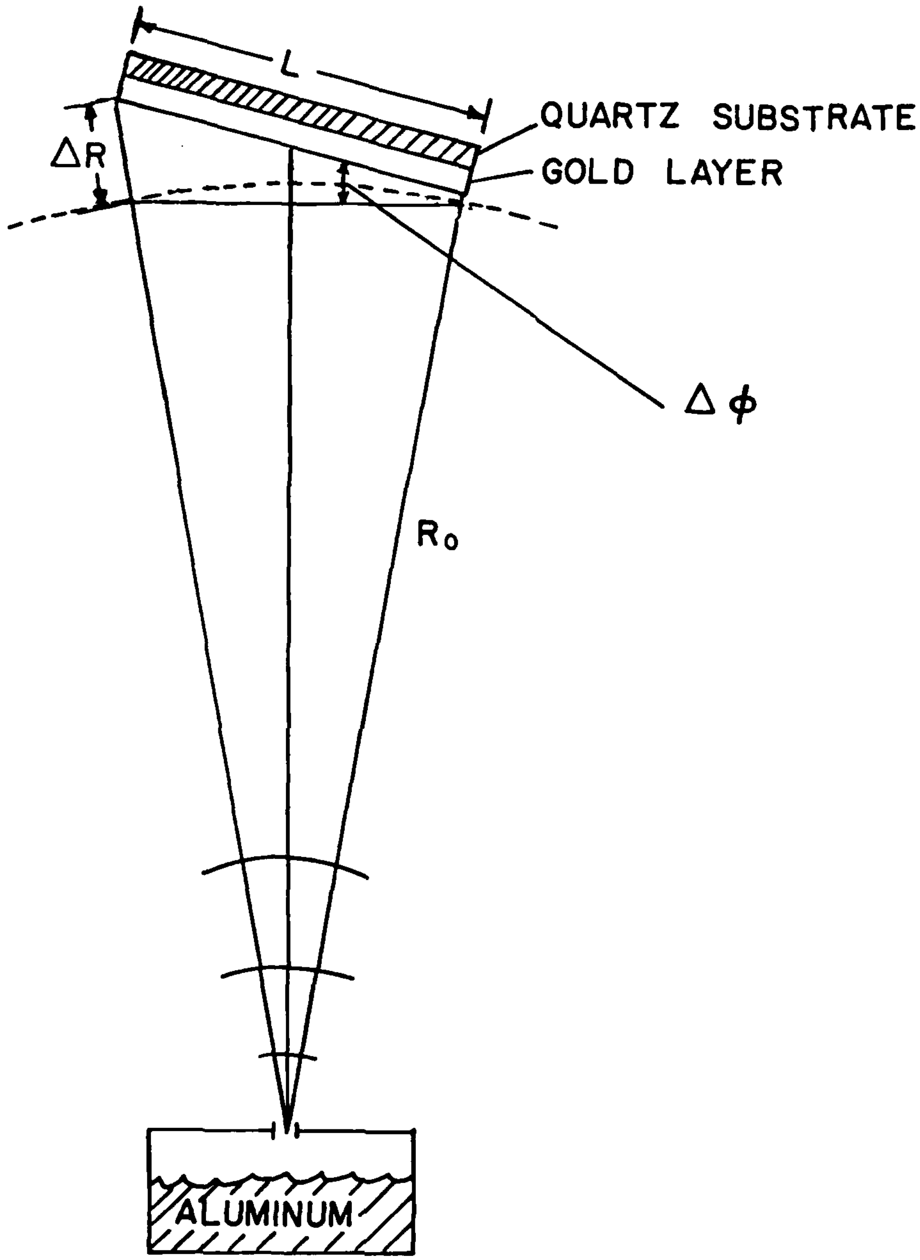

Fig. 4.2. Evaporation technique to produce a wedge in the aluminum layer. The wedge angle $\Delta\phi$, see Fig. 4.1, is directly proportional to the tipping angle $\Delta\theta$.

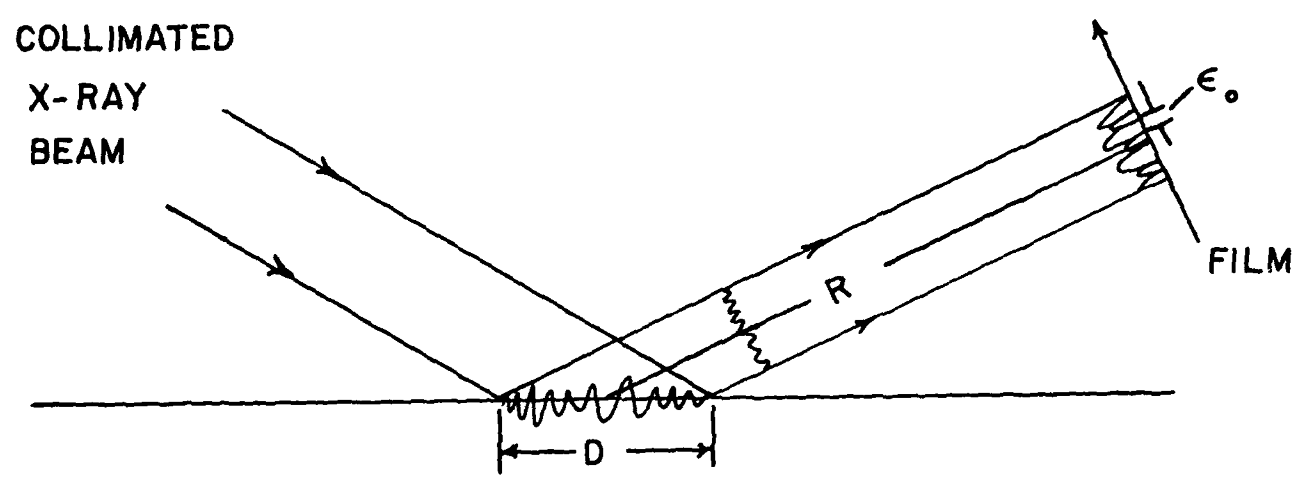

Fig. 4.3. Experimental arrangement for x-ray speckle studies. The speckle size ϵ_o is proportional to $\lambda R/D$, where λ is the x-ray wavelength, D is the spot size, and R is the distance from the rough surface to the observation plane.

The surface can be roughened using a fine grit polish. The speckle pattern is produced by illuminating the roughened surface with a collimated beam of x-rays, and recording the scattered radiation at a distance R from the rough surface. The speckle size, ε_0, at the film plane is proportional to the Airy disc size,

$$\varepsilon_0 \propto \frac{\lambda R}{D} \quad . \tag{4.2}$$

To verify that we are indeed observing speckle, the distance R and spot size D will be varied. If the pattern detail scales as indicated in Eq. (4.2), we can conclude it is speckle.

4.1 Measurement of Film Resolution

In the x-ray speckle experiment, implicitly, we have assumed that the recording media would resolve the individual speckles. This requires only that the distance be large enough that

$$\frac{\lambda}{D} R \gg \text{film resolution.} \tag{4.3}$$

It is in this limit that the granularity, when it is observed, can safely 'e termed speckle. In tests of recording material, the distance R is decreased, and the speckle resolution is checked. When the speckle size, ε_0, is on the order of the film resolution, the speckles should blur or merge.

4.2 Experiments of 20 - 22 May 1980

Several experiments were conducted by Dr. Nicholas George in Dr. Das Gupta's laboratory during the period 20 - 22 May 1980. The special microfocus x-ray apparatus at Texas Technology was used together with some custom-made lead slits. Difficulty was experienced in setting and reproducibility with these slits. Range of gaps used was 1 μm to 10 μm; however, the gap width was not uniform. Several slit-to-film distances were used in the range from 15 cm to 150 cm. The speckle size should vary by 10:1 for this range. This range variation is an effective way to be certain that x-ray mottle of the film is not mistakenly called speckle.

In making the exposures, times ranging from 12 minutes to 240 minutes were used to bring the grey level to a convenient density. Another critical test of speckle was employed. All of the exposures used two tightly clamped pieces of film. The longitudinal size of the speckle is adequate to ensure spatial correlation on the two sheets. X-ray mottle, on the other hand, should not be closely correlated. It is anticipated that this correlation can be conveniently gauged as soon as the negatives are developed. Strong Moiré patterns would be expected if good correlation (or speckle) was present. This correlation was not observed.

Densitometer traces were prepared on each film record upon returning to The Institute of Optics. Figure 4.4 shows a typical result for several distances from slit to film: A (exposure 10 at a distance of 150 cm); B (exposure 8 at 100 cm); and C (film grain noise only). The integrating area used was 3.25×10^{-3} mm^2 which corresponds to 1/4 (B) or 1/16 (A) of the computed speckle size assuming an 0.89 μm slit width. Although both traces show finer structure than the fog level, no structure is observed that scales in proportion to the film-to-slit distances.

Another representative sample is shown in Fig. 4.5 for A (exposure 6 at 50 cm) and B (exposure 8 at 100 cm). In this case the speckle size would double from A to B. Again C is typical grain noise in the film sample.

Both from the lack of spatial correlation on the double film sheets and from a study of the densitometry, as shown in Figs. 4.4 and 4.5, we did not make a definitive observation of speckle. At the time of these experiments, we felt that the major difficulty was with the lack of precision used in setting the slit width. A larger slit width would make a smaller speckle, and thus it would be difficult to observe. For the next series of experiments, we purchased a precision slit and mounted it on a convenient base for the x-ray apparatus.

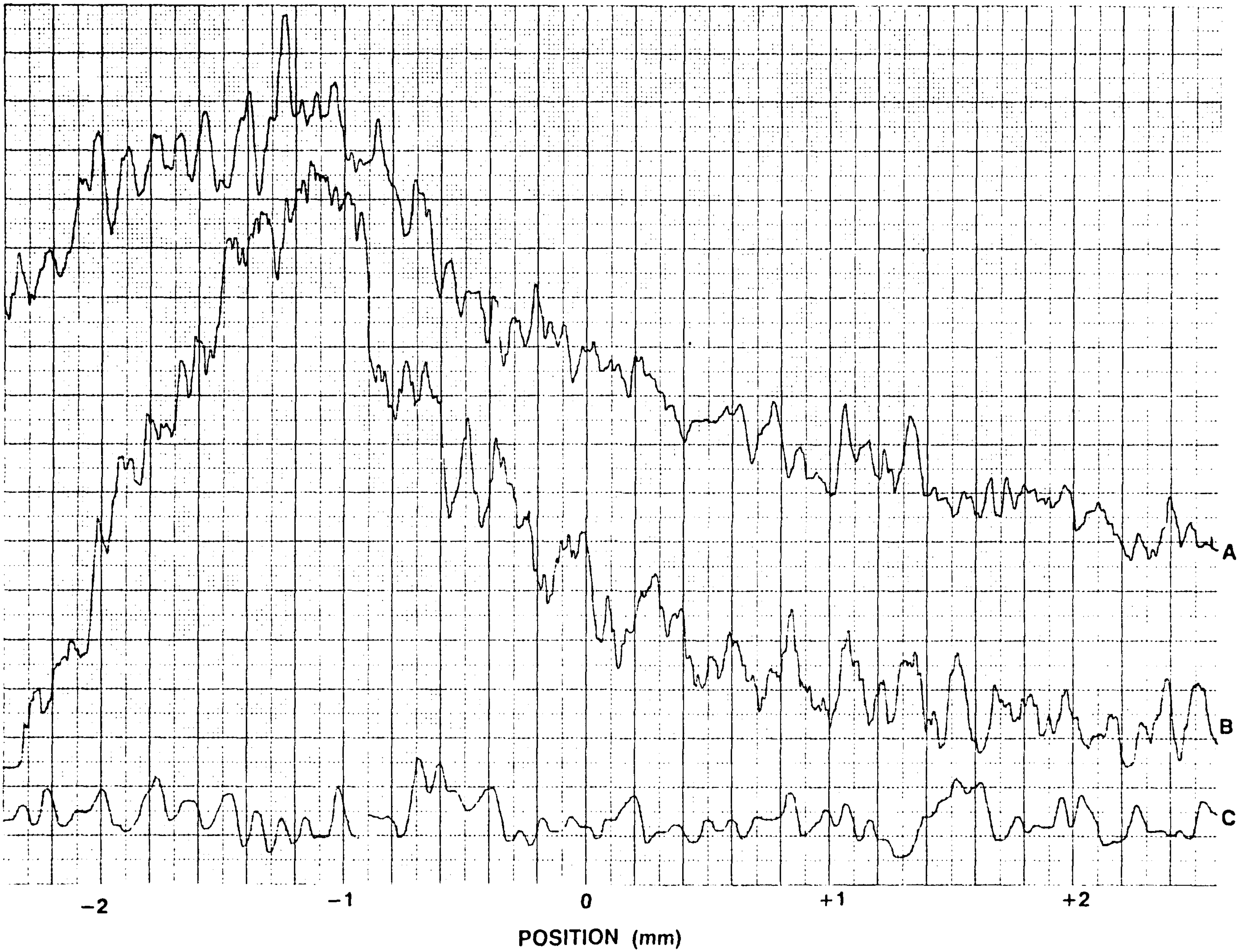

Fig. 4.4. Typical result of speckle test series for 20 - 22 May 1980 experiments: Curve A (150 cm), Curve B (100 cm), and Curve C (Fog level).

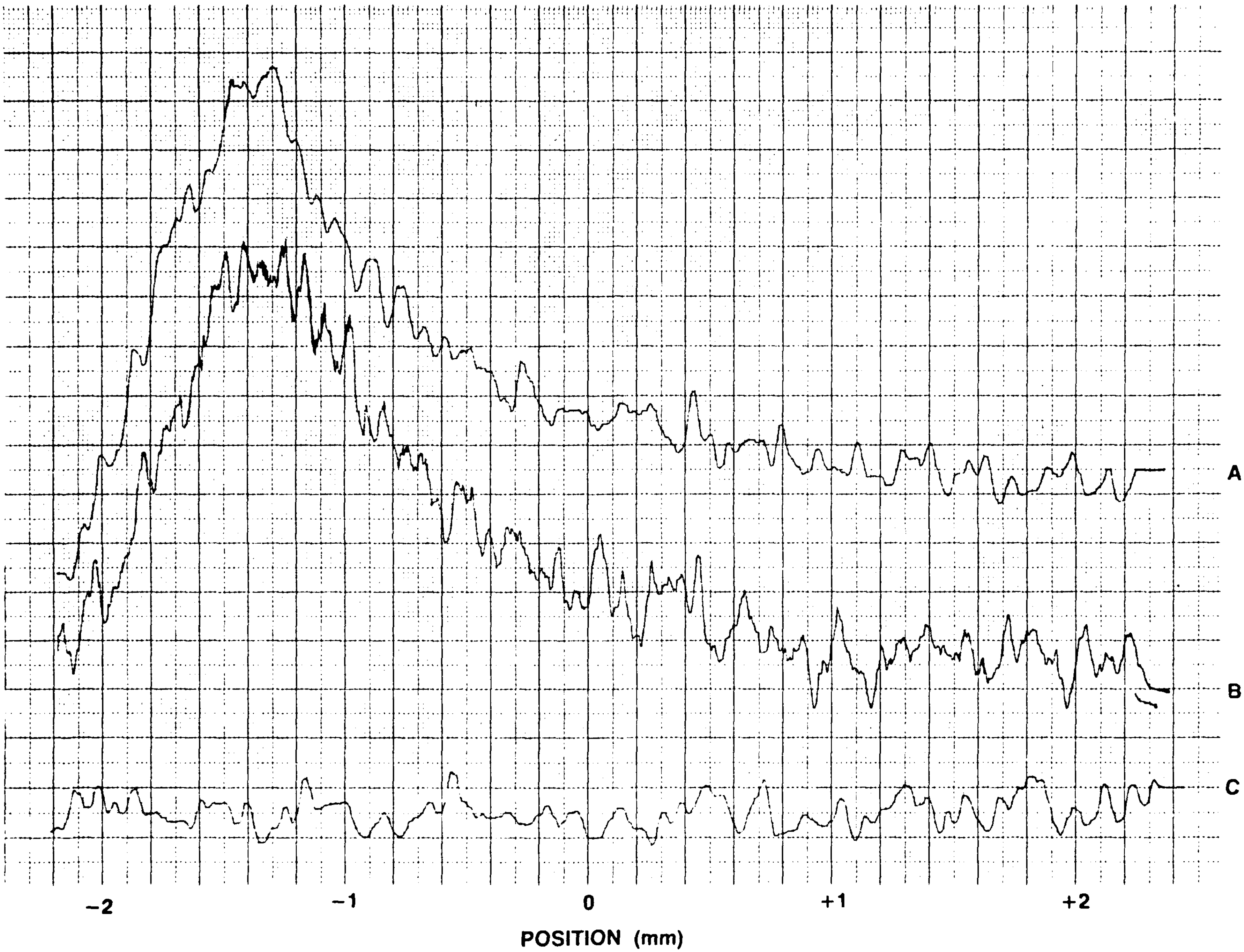

Fig. 4.5. Typical result of speckle test series for 20 - 22 May 1980 experiments: Curve A (50 cm), Curve B (100 cm), and Curve C (Fog level).

4.3 Experiments of 8 September 1980

The objective in these experiments was to observe coherency effects in the x-ray diffraction pattern produced by a narrow slit. Based on our calculations x-ray interference would be observed with slit widths of approximately 1 μm using Professor K. Das Gupta's microfocus x-ray machine. The unique feature of this apparatus is that the source is very small (30 μm x 100 μm). Upon arrival at his laboratory for this second series of experiments, it was found that the high voltage cable, which produces the micro-focus, had broken. The cable could not be repaired, and a new one would have to be fabricated by the manufacturer. The only other machine that was available was a conventional-type x-ray source of dimensions (0.5 mm x 15 mm). With this source it was predicted that the slit width must be less than 0.2 μm in order to observe interference effects. This slit dimension was not possible to obtain using the available equipment.

Nevertheless, it seemed to be worth trying the experiments. The experimental layout is shown in Fig. 4.6. The slit width of 1.1 μm was verified from microdensitometer traces of the diffraction pattern obtained using a He-Ne laser.

Figure 4.7 shows microdensitometer traces of the x-ray pattern recorded at distances A (R = 1.5 m) and B (R = 3.0 m). The trace C is the background (or fog) density. According to diffraction theory, interference lobes should be on the order

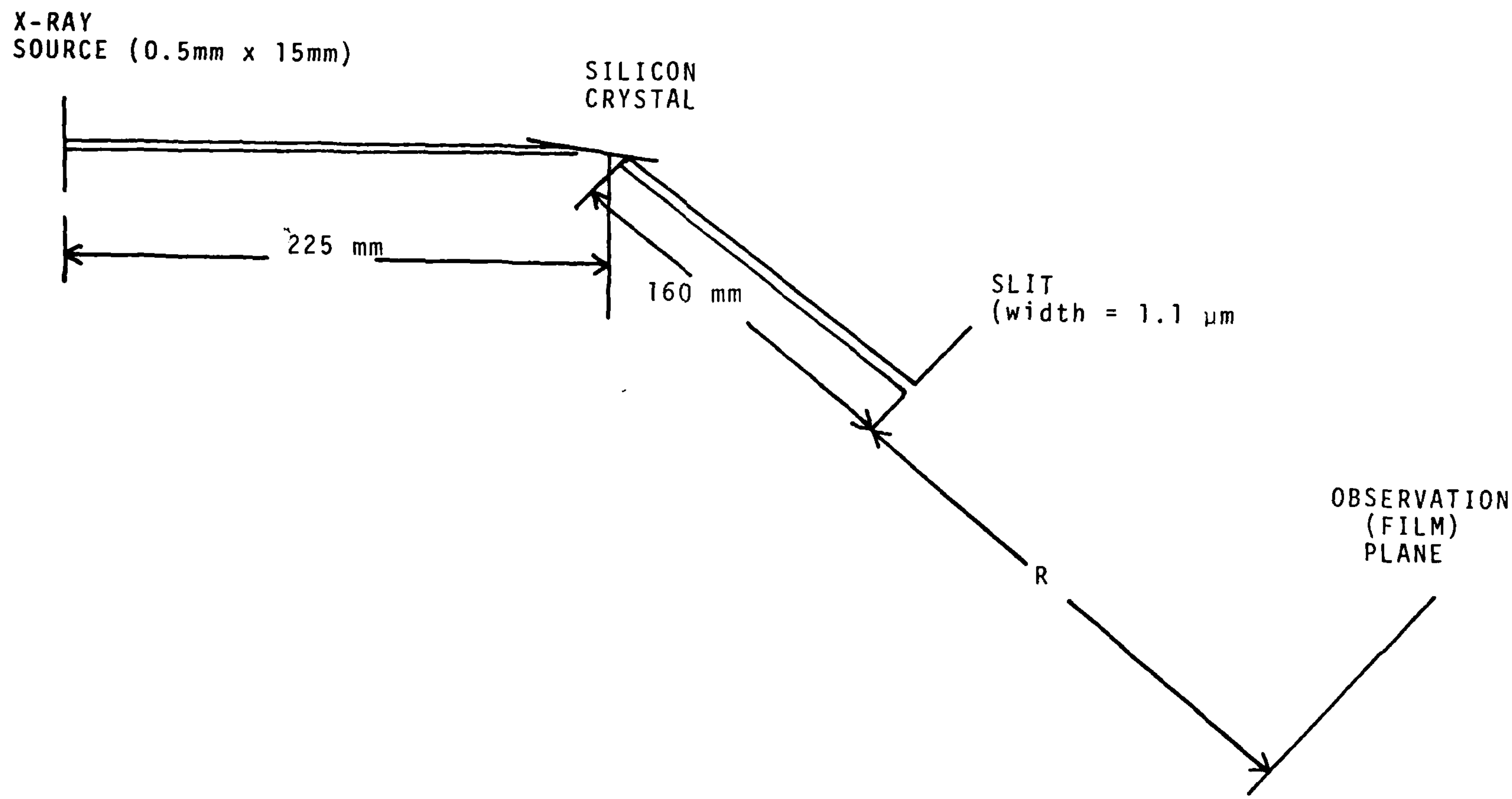

Fig. 4.6. Laboratory layout for x-ray diffraction experiments. R is varied between 50 centimeters and 3.0 meters.

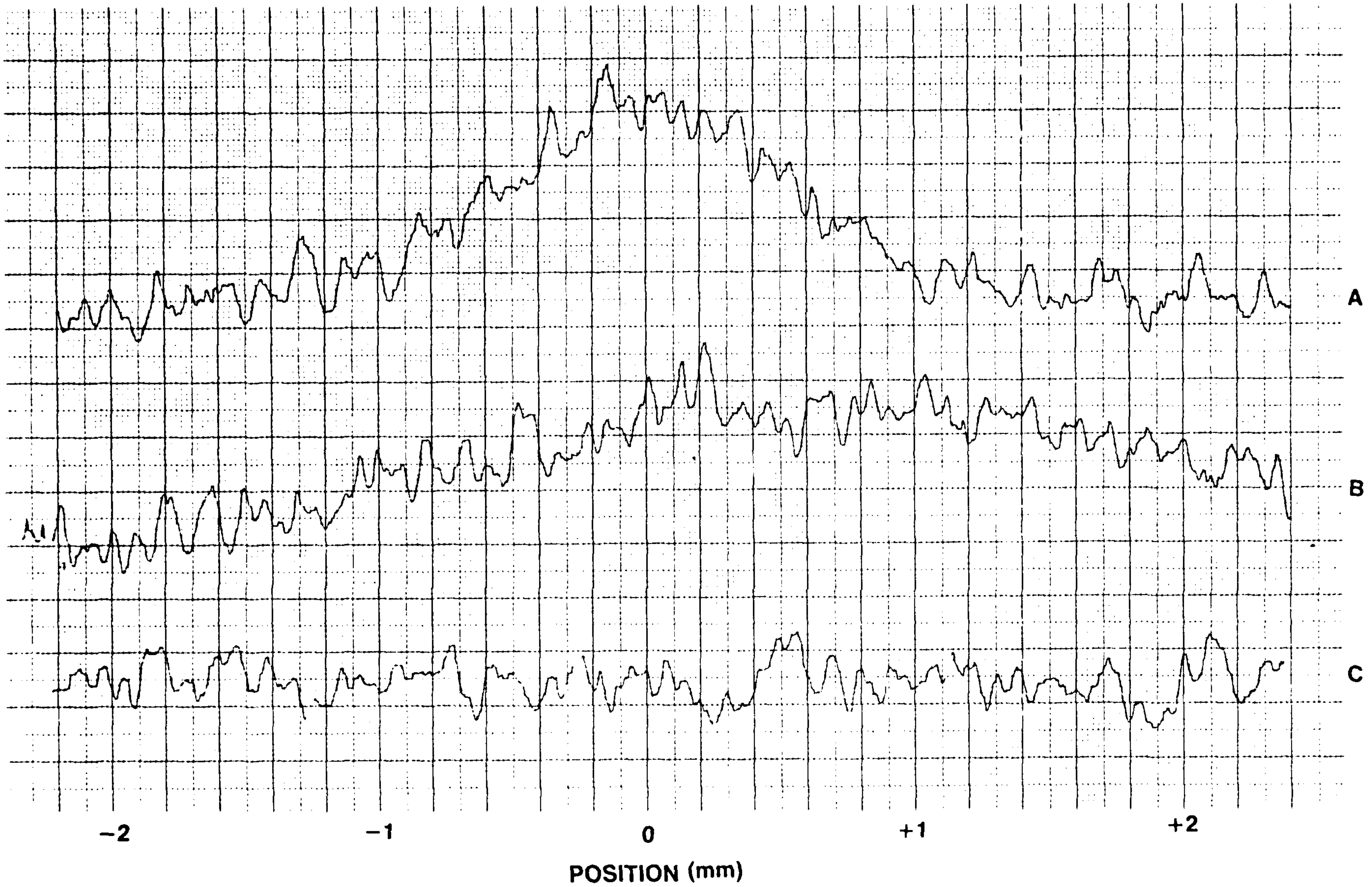

Fig. 4.7. Typical result of speckle test series for 8 September 1980 experiments (Microfocus not operative): A (150 cm), B (300 cm), and C (film background).

of 0.21 mm in (A), and 0.42 mm in (B). This structure is not observed in either trace. Therefore, the conclusion is that speckle phenomena is not observable in these data.

4.4 Conclusions and Plans

One of the major objectives of this research is the observation of speckle from an x-ray source. While at the conclusion of this grant we do not have a definitive series of experiments demonstrating x-ray speckle, it does appear that with some refinement of the experiments in Secs. 4.2 and 4.3 we should be successful in this endeavor. Also the early results in Fig. 2.3 (discussed on p. 6) do constitute a possible measurement of x-ray speckle.

In the more recent experiments, we were plagued with equipment failures of a fairly routine sort. The special apparatus (precision slit and mount, film plate holder, and interference wedge) is in good condition and the x-ray instrument at Texas Technology has been repaired. Arrangements are being made to visit Professor Das Gupta's laboratory during the 1981-82 academic year in order to repeat these experiments. We are appreciative of the funding that was granted by the Air Force Office of Scientific Research for this research. When definitive results have been obtained, this sponsorship will be acknowledged in the resulting publications.

5.0 PERSONNEL

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